Effects of acetone extractives in grey-stage mountain pine beetle-killed lodgepole pine on kraft mills

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Abstract

Previous work has indicated that in a kraft mill pulping a large proportion of grey-stage mountain pine beetle-killed wood, the concentration of extractives in the final bleached pulp increased. As this might give some customers pitch problems and affect product marketability, further research has been done to determine if this is a common problem. New results reveal that, like the wood extractives content in grey-stage pine chips, pulp extractives in grey-stage mill pulps are variable and can occasionally be high (e.g., > 0.05%), likely because of the composition of the extractives in the incoming chips. While this does not usually cause pitch problems in the pulp mill and its customer paper mills most of the time, kraft mills selling grey-stage pulp to extractives-sensitive customers should check the extractives content before shipment as it may occasionally be unacceptably high.

It was also previously reported that the resin acid concentration in the final effluent from this grey-stage mill was high (3 mg/L). As concentrations greater than 1 mg/L could cause toxicity episodes, we have done further testing in several kraft mills using grey-stage beetle-killed wood and found resin acid concentrations to be consistently low and below 1 mg/L.

Keywords: *Pinus contorta*, lodgepole pine, mountain pine beetle, kraft mills, pitch, pitch control, resin, resin acids, fatty acids, sterols, esters, glycerides, tall oil soap, deposits, effluents, extractive content

Résumé

Les travaux précédents ont révélé que dans une fabrique de pâte kraft réduisant en pâte une grande proportion de pins au stade gris tués par le dendroctone du pin ponderosa (DPP), la concentration des produits d'extraction du bois dans la pâte blanchie finale avait augmenté. Comme cette situation serait susceptible d'occasionner des problèmes de poix chez certains clients et nuire à la qualité marchande du produit, une nouvelle recherche a été effectuée, afin de déterminer si cela constituait véritablement un problème. De nouveaux résultats ont révélé que, comme le contenu des produits d'extraction du bois dans les copeaux de pin au stade gris, les produits d'extraction de la pâte dans les pâtes au stade gris des usines de pâtes sont variables et peuvent, de temps à autre, être élevés (p. ex., > 0,05 %), probablement à cause de la composition des produits d'extraction dans les copeaux d'arrivée. Il semblerait que cette situation ne cause habituellement pas de problèmes de poix à l'usine de pâtes et à ses usines de papier clientes, la plupart du temps; cependant, les fabriques de pâte kraft qui vendent de la pâte au stade gris à des clients sensibles aux produits d'extraction du bois devraient vérifier le contenu de produits d'extraction avant l'expédition, car il pourrait être tellement élevé qu'il serait inacceptable.

Il a aussi été relaté précédemment que la concentration d'acide résinique dans l'effluent terminal provenant de cette usine de stade gris était élevée (3 mg/l). Étant donné que les concentrations supérieures à 1 mg/L seraient susceptibles de causer des épisodes de toxicité, nous avons effectué de nouveaux essais dans plusieurs fabriques de pâte kraft en utilisant du bois au stade gris tué par le DPP et nous avons constaté que les concentrations d'acide résinique étaient invariablement basses et inférieures à 1 mg/L.

Mots clés: *Pinus contorta*, pin tordu latifolié, dendroctone du pin ponderosa, fabriques de pâte kraft, poix, contrôle de la poix, résine, acides résiniques, acides gras, stérols, esters, glycérides, tallöl saponifié, dépôts, effluents, contenu de produits d'extraction du bois

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1 Introduction

As mountain pine beetles (*Dendroctonus ponderosae* Hopkins) continue their rampage across British Columbia, infesting an unprecedented number of lodgepole pine (*Pinus contorta* Douglas) trees, they leave behind vast tracts of standing dead grey-stage trees. Our research focuses on the lipophilic extractives in mountain pine beetle-affected wood and their possible implications on issues in kraft mills, such as extractives content of pulp, pitch problems, and effects on effluent treatment (Allen et al. 2009a, b).

The response of lodgepole pine trees to beetle attack (and accompanying blue-stain fungal attack) is to exude canal resin, consisting of resin acids and terpenes, into the bore holes and tunnels made by the beetles. Shrimpton (1972) has observed elevated concentrations of resin acids in wound tissue. Previous work has shown that using red- and grey-stage wood increases resin acid loading to the digester. The resultant decrease in the fatty-acid-to-resin-acid concentration ratio in the black liquor increases the solubility of fatty and resin acids and reduces the yield of soap skimmings (Uloth et al. 2007). The increased extractives load that often accompanies use of beetle-affected wood has to be removed in brownstock washing for good pitch control. Across the bleach plant and at the pulp machine, using green-attack and red-attack wood did not significantly change the normal quantities of extractives in pulp (Allen et al. 2009a).

At the time of sampling for this previous work, only one kraft mill in British Columbia was using predominantly grey-stage beetle-affected lodgepole pine. In this mill, the quantity of extractives, especially the unsaponifiables, in the final pulp was significantly higher than normal (Figure 1). Moreover, the resin acid concentration in the final mill effluent was high. These observations were inconsistent with general mill experience. We felt that the results from the grey-stage mill may have been anomalous and so would require confirmation by conducting trials in other mills.

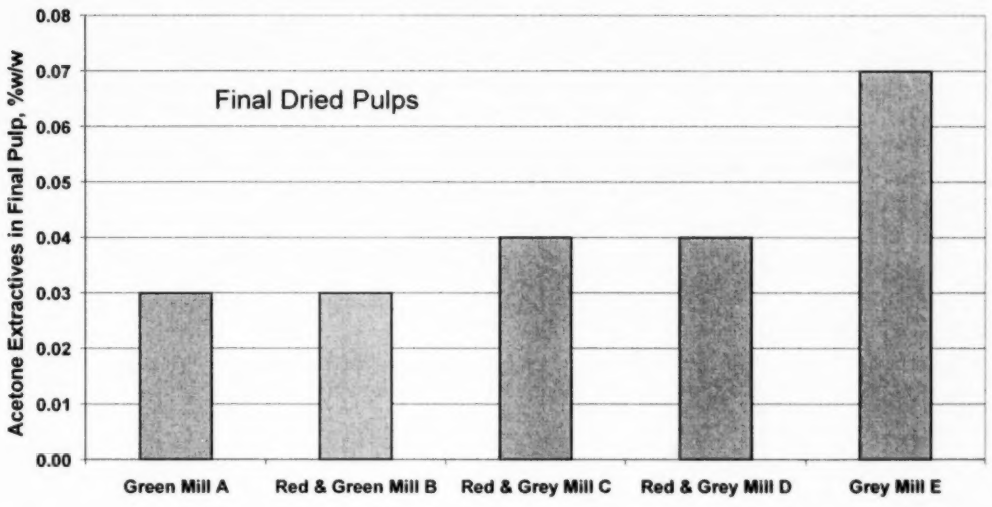

Figure 1. Acetone extractives of final pulps from the mills studied in previous work (Allen et al. 2009a). The one grey-stage mill had a significantly higher extractives concentration than the others.

The objective of this study was to determine whether using grey-stage beetle-killed wood increases concentrations of acetone extractives in the final bleached pulp and increases resin acid concentrations in the treated effluents.

During the time between this and the previous study, more British Columbia mills had begun using mostly grey-stage beetle-killed wood.

2 Materials and Methods

The collection of composite samples at the mills and their subsequent analyses were described in Allen et al. (2009a, b). Further samples were taken from Mill E and Mill D (which was using predominantly grey-stage beetle-killed pine).

In a separate experiment, we did two runs in our pilot plant pulping and bleaching facility, one with unaffected wood and the other with grey-stage chips. The unaffected wood was from a wet site and had an acetone extractives content of 5.2%. The grey-stage chips were from a moist site and had an acetone extractives content of 5.0%. We estimated that the trees had been killed by the beetles eight years earlier. Standard cooking and bleaching conditions were used, following the conditions used in Mill E. After cooking, oxygen delignification, and all the bleaching stages, the pulps were thoroughly washed (hyper-washed) because we felt that the results would be significant since both the unaffected and grey-stage chips would be processed the same way.

Conditions for the pilot plant cooks are given in Table 1. Pilot plant cooks were done with the unaffected and grey-stage chips in a 20 L digester simulating LoSolids® conditions at 14% effective alkali concentration, 30% sulphidity, and a 5:1 liquor-to-wood ratio (including chip moisture), as described by Radiotis et al. (2001).

Bleaching conditions and related parameters are given in Table 2.

Table 1. Conditions of Kraft Cooks

Impregnation Vessel	
White liquor split, % of total	50
Temperature, °C	110
Retention time, min	30
Upper Cooking Zone	
White liquor split, % of total	0
Temperature, °C	165
Retention time, min	10
Lower Cooking Zone	
White liquor split, % of total	50
Temperature, °C	165
Retention time, min	195
Wash Circulation	
White liquor split, % of total	0
Temperature, °C	165
Retention time, min	80

Table 2. ODEopDEpD Bleaching Parameters.

	Unattacked Chips	Grey-Stage Chips
O_2 Delignification: (conditions: 95°C; 60 min; 10% cs.[1]; 517 kPa [75 psig])		
NaOH, % on o.d. pulp	1.70	1.70
$MgSO_4$, % on o.d. pulp	0	0
Final pH	10.7	10.5
Kappa number	16.8	15.2
Delignification, %	42.9	46.5
D_0 Stage: (conditions: 55°C; 30 min; 2.8% cs.)		
ACM	0.170	0.170
ClO_2, % on o.d. pulp	1.09	0.983
ClO_2, % uptake	1.07	0.973
Final pH	3.05	3.09
Eop Stage: (conditions: 80°C; 20 psig for 8 min; 60 min total; 10.8% cs.)		
NaOH, % on o.d. pulp	2.30	2.30
H_2O_2, % on o.d. pulp	0.44	0.44
H_2O_2, % uptake	0.44	0.44
$MgSO_4$, % on o.d. pulp	0	0
Final pH	11.8	11.9
Kappa number	4.4	4.2
D_1 Stage: (conditions: 75°C; 120 min; 11.0% cs.)		
ClO_2, % on o.d. pulp	0.97	0.97
ClO_2, % uptake	0.97	0.97
NaOH, % on o.d. pulp	0.40	0.40
Final pH	3.6 at 30°C	3.6 at 30°C
Brightness, % ISO	86.6	86.0
E_2p Stage: (conditions: 80°C; 55 min total; 10.5% cs.)		
NaOH, % on o.d. pulp	0.70	0.70
H_2O_2, % on o.d. pulp	0.22	0.22
H_2O_2, % uptake	0.19	0.21
Final pH	11.3 at 40°C	11.3 at 40°C
D_2 Stage: (conditions: 75°C; 150 min; 11.0% cs.)		
ClO_2, % on o.d. pulp	0.20	0.20
ClO_2, % uptake	0.18	0.18
NaOH, % on o.d. pulp	0.00	0.00
Final pH	4.2 at 40°C	4.2 at 40°C
Brightness, % ISO	91.7	91.3

[1] pulp consistency

3 Results and Discussion

3.1 Acetone Extractives at Key Points along the Process

Figure 2 shows the acetone extractives at various stages in the process for three mills. Mills D and E also have results from the present set of samplings, labelled as "later." For these later samplings, a profile of extractives through the mill was generated, and samples were taken of the final pulp 1 and 2 weeks after the profile sampling. To put the latest results in perspective, we have included the previous work from Mills B, D, and E. (Mills B and D do not have oxygen delignification and, consequently, have no samples from the pre-oxygen washer.)

For the bar graphs labelled Mill D (later), the extractives contents of the pulps tend to be a little higher across the mill than in the earlier sampling (Mill D), especially at the D_0 and D_1 stages, and pulp machine headbox. Nevertheless, these results are not higher than those for Mill B, which was using predominantly green- and red-stage beetle-affected wood.

In the five extractives profiles in Figure 2, it is evident that the results from the D_0 stage of bleaching and pulp machine headbox are consistently higher than the preceding sampling point. This can be true of the D_1 and D_2 stages, but not consistently. One reason for the higher extractives content in the D_0 stage, and why they are higher than the preceding sample in the fibre line, is that the low pH (< 3) causes the dispersed wood resin particles and dissolved soaps to coagulate on the fibre surfaces (Allen and Lapointe 1987). They are subsequently re-dispersed in the Eop stage and so can be washed readily from the pulp. Depending on the pH, this can also happen in the D_1 and D_2 bleaching stages. A second reason for the increased extractives content in the D stages is that chlorine dioxide partially chlorinates the resin, which increases its molecular weight to raise extractives content. The pulp machine headbox samples show higher extractives contents due to its white water system's intense recirculation that concentrates the dispersed extractives (Ricard and Dorris 2007).

For Mill D (later), despite the higher extractives content in the profile, the final pulp extractives content is low and < 0.05%. This is also true for the later samplings at Mill D after 1 and 2 weeks.

For Mill E, the later samplings (Figure 2, bottom bar graph) were lower in extractives right across the mill, despite higher pre-oxygen washer extractives content. This contrasts sharply to earlier Mill E results in the second-last bar graph. In our new round of sampling, the extractives content of the final pulps was consistently low and < 0.05%, so we surmise that the earlier result for Mill E was unusual.

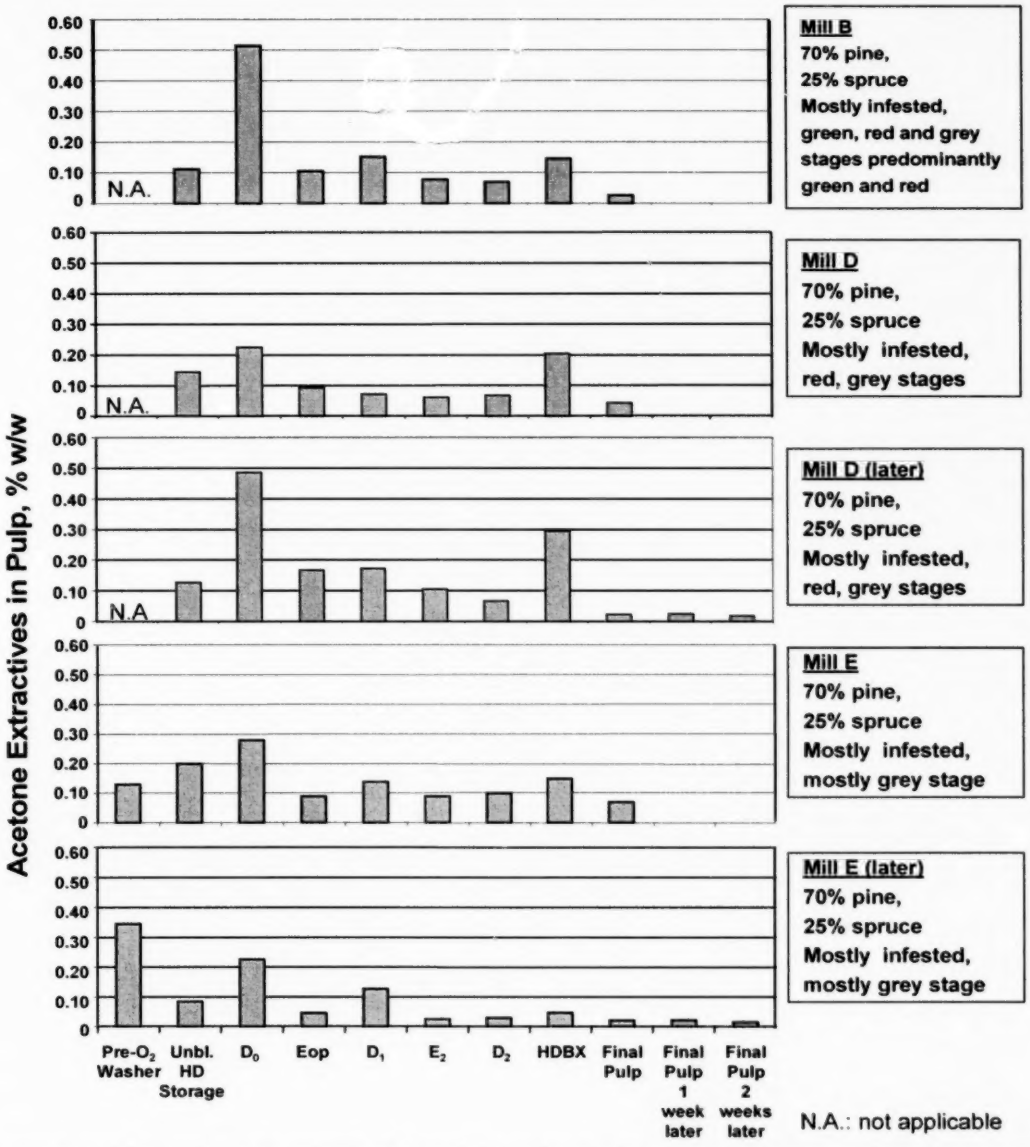

Figure 2. Acetone extractives content (% by weight on dry pulp) versus sampling point along the process stream.

Note: As in previous work [1], the mills have been arranged so that the time of infestation of the wood used increases in proceeding from top to bottom. In the cases of Mills D and E, results from the most recent sampling are shown in the third-from-bottom and bottom bar graphs, respectively. For comparison, earlier results from Mills B, D, and E are also shown.

Extractives content varied considerably, much of it likely from variations in chip extractives (Figure 2). Examples of this include extractives in the final pulps [Mill D and Mill D (later), and Mill E and Mill E (later)]. The variations in extractives content are also evident in the unbleached high density storage pulps (both mill-to-mill and within-mill).

The range of acetone extractives content for both unattacked and beetle-killed lodgepole pine is 1.5%–6.0% (Bicho et al. 2007). Consider the physiology of the extractives and why they do not advance to even higher amounts in chips from beetle-attacked trees. Softwoods have two kinds of wood resin: canal resin (consisting of terpenes and resin acids) and parenchyma (ray) cell resin (consisting of fatty and resin acids, glycerides, sterols, steryl esters, and wax esters). Canal resin is synthesized in special parenchyma cells that line the resin canals. It is under several atmospheres of pressure and flows to the site of attack when a beetle enters the bark. In most trees, this is followed by a massive production of more canal resin (secondary resinosis). Extractives levels, especially of resin acids but also of other extractives, increase. After the beetle attack, blue-stain fungus infects the tree, using fatty acids, steryl esters, and glycerides as food. This keeps the extractives content from rising much above 6%, but profoundly alters resin composition so that the fatty-to-resin acids ratio is greatly lowered. Then, the resins are slowly oxidized to CO_2 and water. In pulp mills using sawmill chips, the chips are mainly from sapwood, which has a lower extractives content. Some mills are now using chips from roundwood, which increases extractives content. Removing bark from roundwood drains resin acids from the area just under the bark where the beetles are active. Chip washing and screening further flushes resin acids.

3.2 Acetone Extractives of Pilot Plant Pulps

Figure 3 gives the acetone extractives content for the two pilot plant pulps. Since pulp samples were thoroughly washed prior to extraction, the increases in extractives at the D_0 and D_1 stages are almost certainly attributable to chlorination (chlorine atoms and hydroxyl groups are added at some of the double bonds, which increases the resin's molecular weight and hence the extractives content).

Pulps derived from grey-stage chips consistently have higher extractives contents from the unbleached pulp through to the D_2 (final) pulp stages, despite unattacked and grey-stage chips having approximately the same extractives content (5.2% and 5.0% respectively). The extractives content of the final grey-stage pulp is over 0.12%, which is much greater than the 0.05% maximum normally encountered in well-run northern bleached softwood kraft mills with reasonably good equipment. This observation contrasts with mill results in Figure 2, with the exception of Mill E. The chemical composition of the extractives is the likely reason for their higher content in grey-stage pulp, as will be explained later in our discussion of Figure 5.

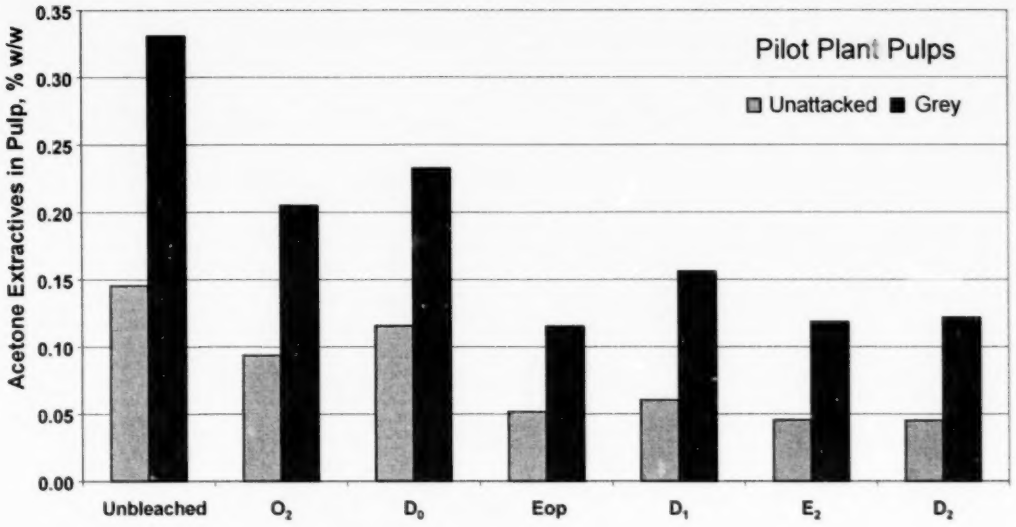

Figure 3. Acetone extractives content (% by weight on dry pulp) of pilot plant pulps at each stage in the manufacturing process.

Note: Unlike the recent mill-sampled results in the previous figure, this grey-stage pulp had significantly higher acetone extractives content throughout the process and in the final pulp. In this respect, it resembled the earlier results for Mill E.

3.3 Composition of Acetone Extractives of Mill Pulps

Figure 4 shows the composition of five acetone extractives profiles at various points along the process stream, arranged as in Figure 2. Mill E has higher concentrations of sterols than Mill E (later), especially after the Eop bleaching stage and downstream. The ratio of saponifiables (fatty and resin acids) to neutrals (sterols) is lower throughout Mill E. Mill D and Mill D (later) have similar extractives compositions in the above respects to Mill E (later). These results indicate that the higher saponifiables-to-unsaponifiables ratio in the extractives of Mill D, Mill D (later), and Mill E (later) are advantageous for good deresination, which fits well with the prevailing understanding that fatty acids can form sodium soaps in alkaline conditions that help remove unsaponifiable extractives by forming micelles and solubilizing (Ström 2000, Allen 2000).

The variation in the final pulps' extractives content in Figure 2 is more likely due to the extractives' composition rather than to variations in the wood's extractives content. For example, samples of pulp (after Eop) with the highest saponifiables-to-unsaponifiables ratio in Figure 4 generally led to the lowest final pulp extractives content in Figure 2.

As in our previous publication (Allen et al. 2009a), results in Figure 4 are presented as mg/g of extract. Gas chromatographic yields for all samples are higher before the D_0 stage of bleaching. This is largely because of peak identification issues in our gas chromatographic analyses, but may also be partly explained by more oxidation and polymerisation on the fibre line in the D_0 and later stages.

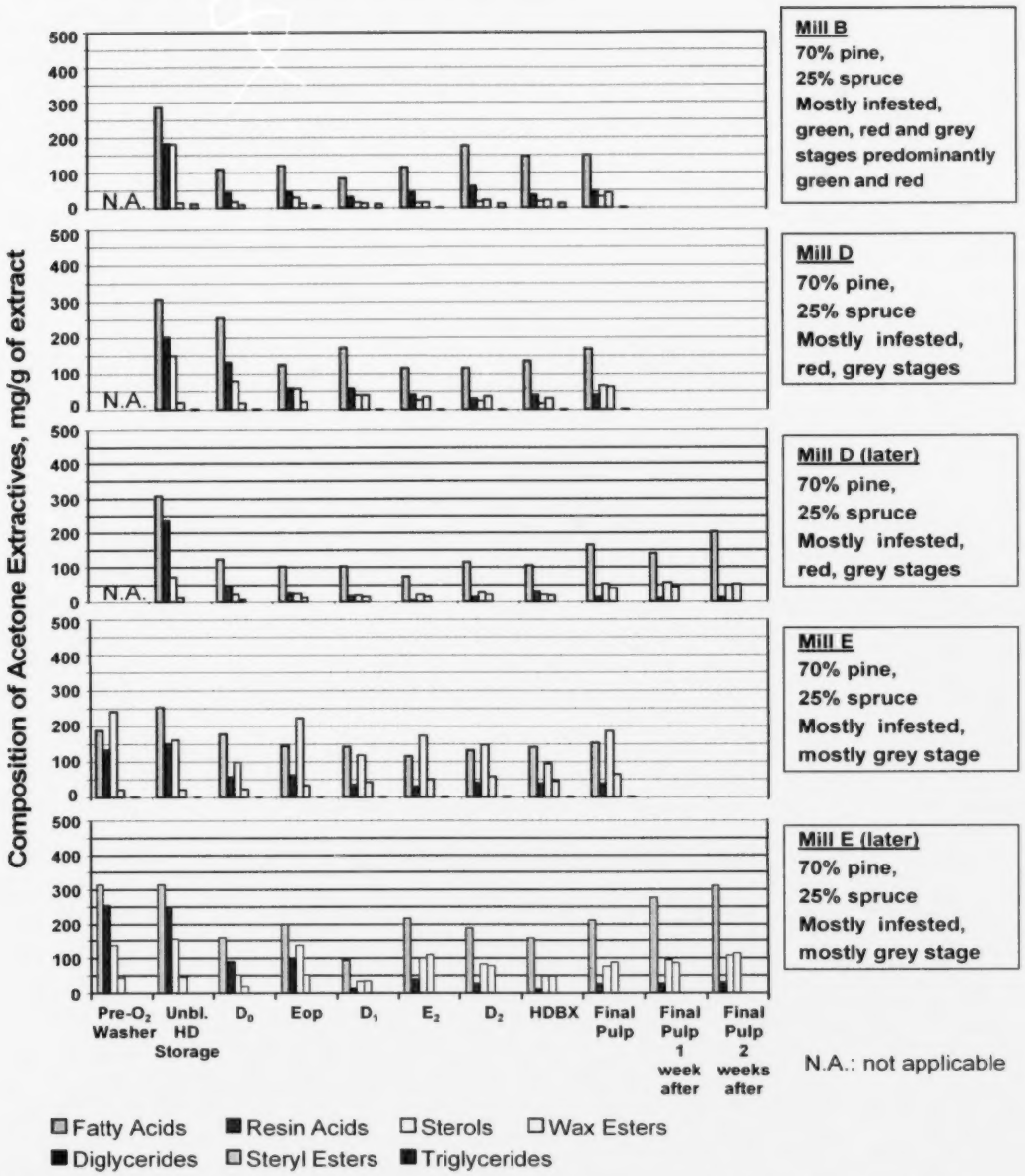

Figure 4. Composition of the acetone extractives versus sampling point along the process stream.

Note: As in Figure 2, infestation time increases from top to bottom; results are presented for the same samples as in Figure 2. Samples from the later round of sampling (third and bottom bar charts) differ considerably in composition from the previous samples for Mill E (second-last chart), which showed larger concentrations of sterols and a lower ratio of saponifiables-to-unsaponifiables.

3.4 Composition of Acetone Extractives of Pilot Plant Pulps

Figure 5 shows the composition of the acetone extractives in the pilot plant pulps, with results for pulps from unattacked and grey-stage chips on the left and right respectively. The two pulps have markedly different extractives compositions: grey-stage pulps tend to have less fatty acids and more sterols, as was also evident in the Mill E pulp (Figure 4). Also, there are more resin acids in the unbleached grey-stage pulps up to the Eop stage, which was not evident in Figure 4. As the results in Figure 4 indicate, the lower saponifiables-to-unsaponifiables ratios of grey-stage pulps appear to lead to higher extractives contents in the final pulps, seen previously in Figure 3.

All triglycerides and diglycerides hydrolysed (saponified) completely during the cooks, as was seen in the mills (Figure 4). This time, there were no steryl esters in the grey-stage pulps, as there were in trace quantities in the high extractives final pulp of Mill E, seen in Figures 2 and 4.

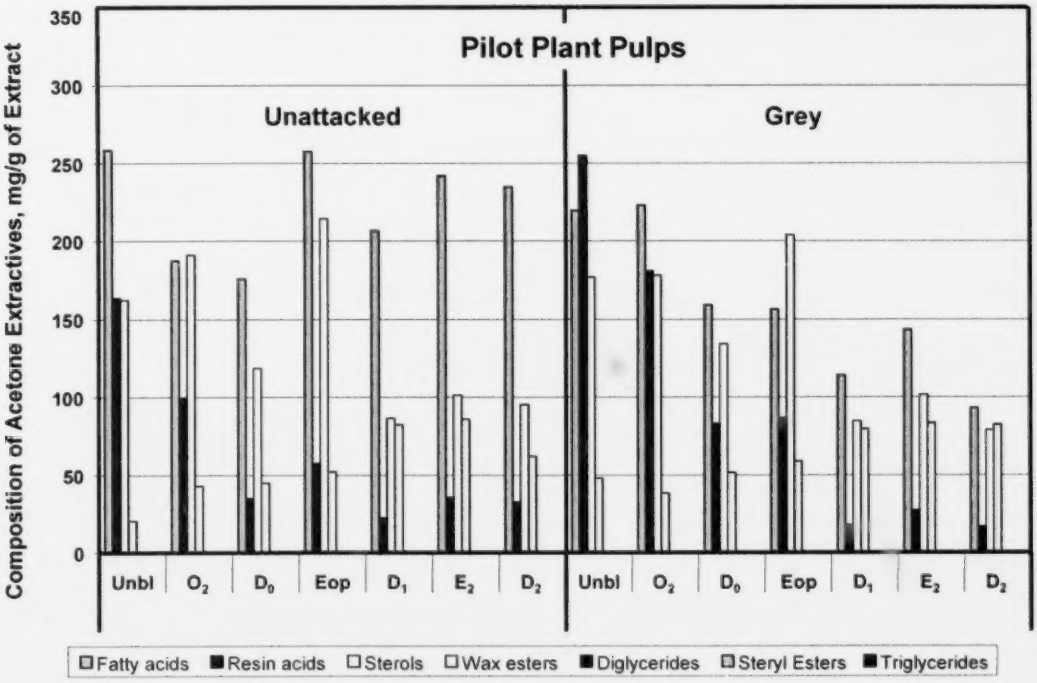

Figure 5. Composition of the acetone extractives of the pilot plant pulps.

Note: Compositions of extractives in the grey-stage pulps are closer to those of Mill E (original sampling: Figure 4, second-last graph), in that the saponifiables-to-unsaponifiables ratio is lower.

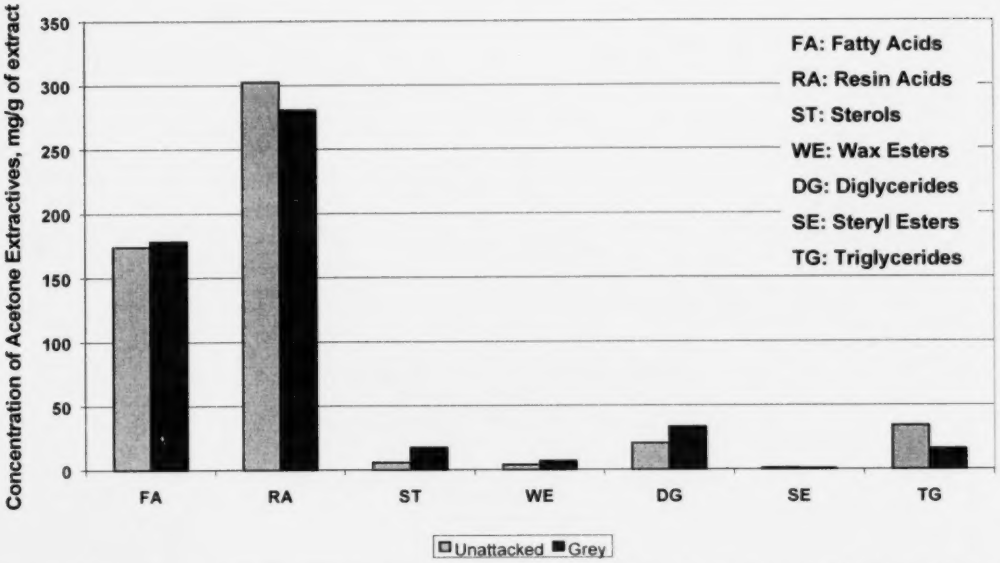

Figure 6. Composition of acetone extractives of unattacked and grey-stage wood chips used in pilot plant experiments.

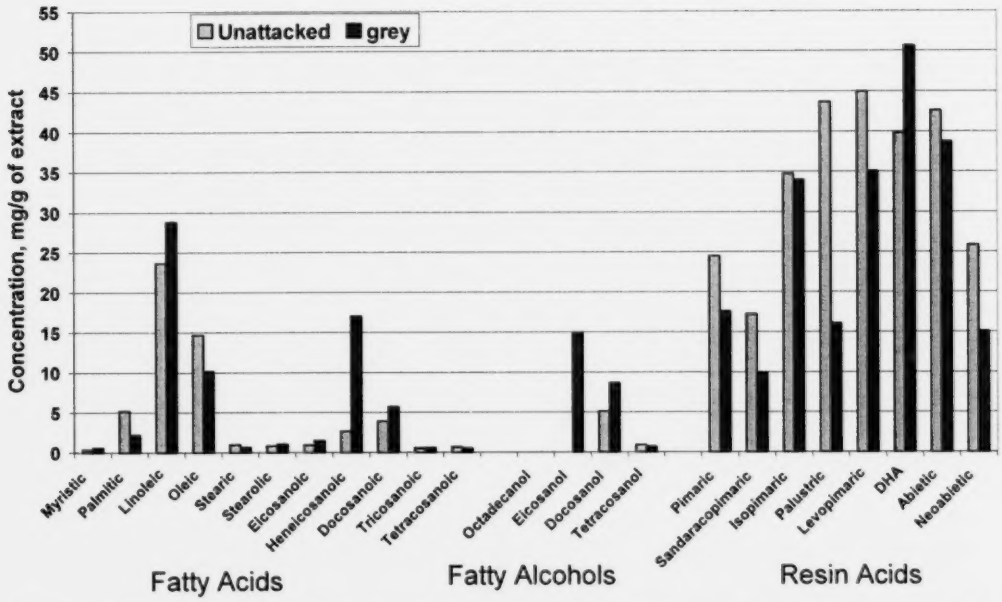

Figure 7. Detailed characterisation of components in the fatty and resin acid fractions of the acetone extractives of unattacked and grey-stage wood chips used in pilot plant experiments. The fatty alcohols in this graph were included as fatty acids in Figure 6.

The higher extractives in the final (D_2) pilot plant pulps provide an opportunity to look at the chemical composition of the higher extractives final pulps occasionally associated with the use of grey-stage wood. The compositions of the extractives in the chips from the unattacked and grey-stage woods are shown in Figure 6. The fatty and resin acid contents are remarkably similar; however, the concentrations of sterols and wax esters are higher in the grey-stage chips. Figure 7 shows a full chemical analysis of the fatty and resin acids. It is clear that, whereas the total amounts of fatty acids are about the same (Figure 6), the compositions of the unattacked and grey-stage wood differ considerably. The latter (Figure 7) shows more high molecular weight fatty acids (especially C_{21} and C_{22}) and an unexpectedly high amount of fatty alcohols. As these are unusual in softwoods, we hypothesise that they may be associated with the blue stain fungus.

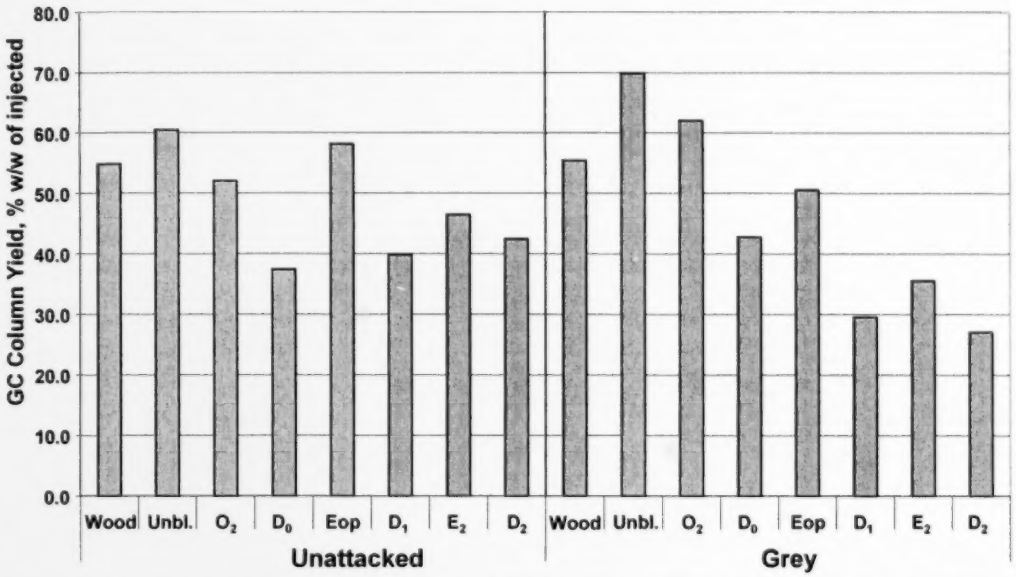

Figure 8. Components amenable to GC analysis in acetone extractives of unattacked and grey-stage pulps in pilot plant experiments (shown in Figure 6).

Another possible difference between the unattacked and grey-stage wood is the extractives' degree of polymerization. Those in seasoned wood (especially the fatty acids and glycerides) can polymerize to give higher molecular weight material (Sitholé et al. 2008, 2009). Ordinarily, high molecular weight polymerised extractives are not soluble in solvents such as acetone, but intermediate molecular weight polymerized material is somewhat soluble, hence they can add extractives to the final pulp even though high molecular weight material cannot. A measure of the intermediate molecular weight extractives can often be obtained from the yield of the gas chromatograph column in analyses, such as those shown in Figure 6. Intermediate molecular weight extractives usually do not elute.

In Figure 8, column yield is plotted for the pilot plant pulps. Yields from unattacked and grey-stage chips are about the same, which suggests that intermediate molecular weight polymerized extractives do not significantly contribute to the difference in the amounts of extractives in the final pulps. After the Eop stage, the yields of the grey-stage pulps diminished to about two-thirds of those of the unattacked wood for D_2; this may be attributable to greater chlorination after Eop of the C_{21} and C_{22} fatty acids and fatty alcohols observed in Figure 7. (The C_{20}–C_{24} fatty acids are labelled as saturated

in Figure 8, but the analysis does not differentiate saturated from unsaturated and most are likely unsaturated.)

The high extractives occasionally observed in kraft mills pulping grey-stage chips (and also observed in our pilot plant experiment) may also be attributable to extractives associated with blue-stain fungus, which take up more chlorine in the bleach plant.

3.5 Composition of Acetone Extractives of Sewered Waters

Figure 9 shows the chemical compositions of the extractives in sewered waters at the primary clarifier, the secondary treatment system, and in the final effluent. Mill D (later) and Mill E (later) samples were taken one and two weeks after the initial sampling. Most of the bars have a red and blue portion, indicating the contribution of the water phase and solid phase of the effluent, respectively.

Mill E had an unusually high concentration of both resin acids and sterols in the final effluent (Figure 9) (Allen et al. 2009a). These high values are not evident in samples from Mill D (later) and Mill E (later), nor are they evident in these two mills one and two weeks after the initial profiling. In all the latest rounds of sampling, the resin acid concentrations were < 1 mg/L. Concentrations of sterols were low. All this suggests that the previous results of Mill E are not representative of effluents of kraft mills using grey-stage wood.

The apparent high values in the final effluent of Mill E (later) two weeks after the main sampling (especially the fatty acids and sterols), were found to be attributable to silicone oil. (There were extra peaks that could not be differentiated by gas chromatography—flame ionization detection; however, additional analyses indicated silicone oil.) The oil was likely due to brief use of a defoamer at the mill to quell foam in the effluent treatment system.

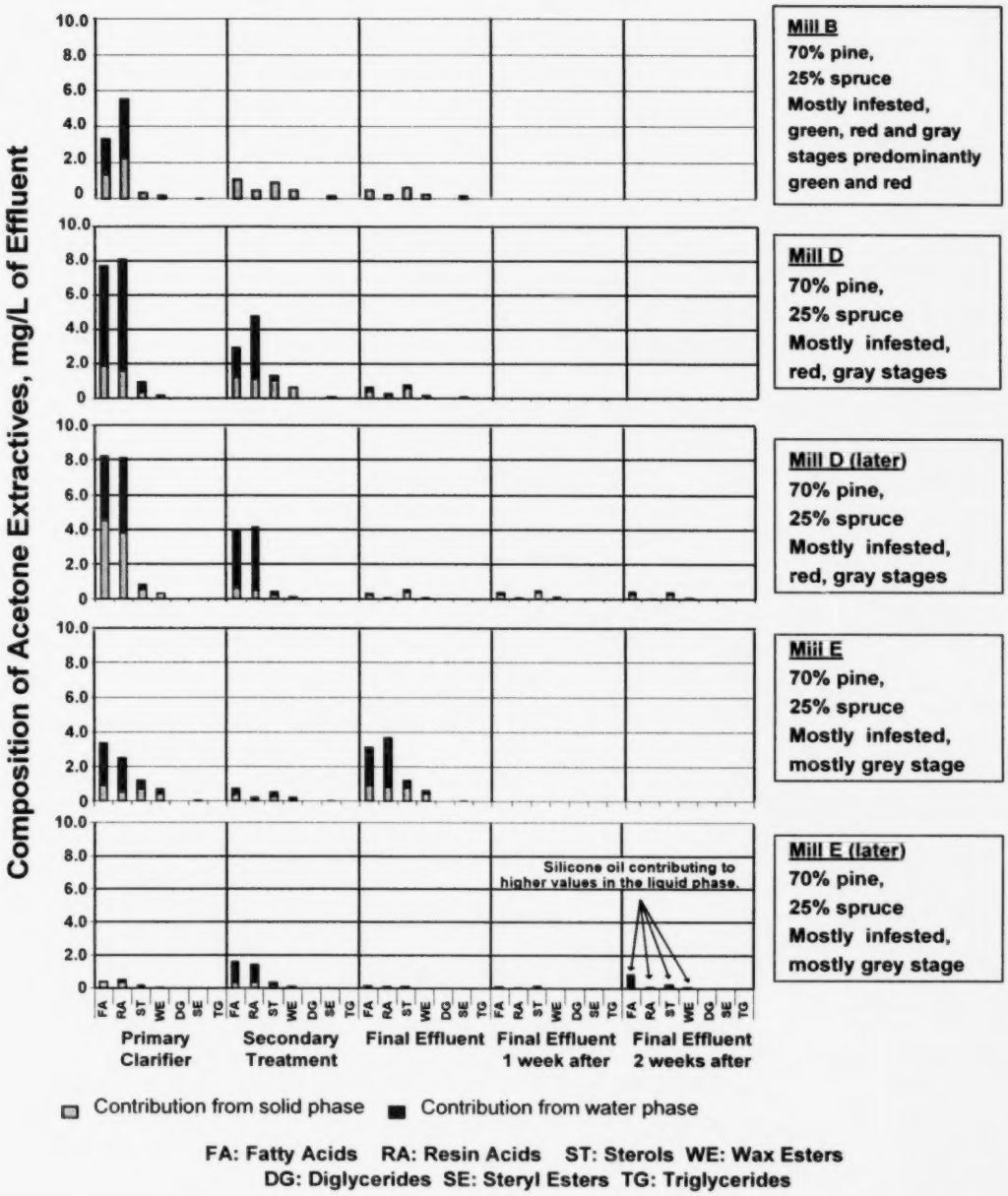

Figure 9. Acetone extractives of solid and aqueous phases of sewered water to primary clarifier, secondary treatment, and final outfall.

Note: Each bar shows results from the solid phase (blue) and water phase (red). Unlike the original effluent sample from Mill E (second-last bar graph), all subsequent samples had low concentrations of resin acids and sterols in the final effluent.

4 Conclusions

Extractives contents of the final pulps produced from grey-stage beetle-affected chips vary and can be high. Mill experience and our results suggest that this is rarely a problem. In kraft pulp mills using mostly grey-stage beetle-killed wood, the extractives content of the incoming chips can vary greatly from 1.5% to 6% (Bicho et al. 2007). High extractives present a greater challenge for deresination during brownstock washing, and it is important to adhere to good pitch control practices, such as avoiding bypassing brownstock washers, good white liquor clarification, high temperature of initial brownstock washing, and judicious use of defoamers (Allen 2000).

Most paper mills will be unaffected by occasional increases in the extractives content of kraft pulp. However, some paper mills operate such that they are continually on the edge of a pitch problem. In other paper mills, the final paper must meet low extractives specifications. Kraft mills selling grey-stage pulp to such extractives-sensitive customers should check the extractives content of their pulp before shipment, as it may sometimes be unacceptably high.

Our results suggest that the reason for the occasional high extractives content of final pulps from kraft mills using grey-stage beetle-killed wood appears to lie more in the chemical composition of the extractives than in their amounts in the incoming wood. The elevated resin acid content of beetle-killed wood results in a lower fatty-to-resin acid ratio in kraft black liquor, and the result is greater solubility of resin and fatty acids in the liquor. Although this gives poor soap skimming, it does mean that the resin and fatty acids, along with some of the other extractives (through a phenomenon called solubilization), are more easily washed from the pulp in the unbleached washers. So the high lodgepole pine chip extractives rarely result in high extractives in the final pulp. The occasional high extractives content in grey-stage lodgepole pine pulps appears to be associated with a high sterols concentration in the chips and extensive blue-stain fungal attack. Because of the good fatty and resin acid removal in brownstock washing, the resultant lower saponifiables-to-unsaponifiables ratio of the extractives in the bleached pulp downstream of the Eop washer causes poor deresination in later stages of bleaching and washers. This may be exacerbated by a higher tendency of extractives associated with blue-stain fungus to take up chlorine on the double bonds.

In effluents from mills using grey-stage beetle-killed wood, we found no further evidence that resin acid concentrations can be high. Indeed, all of the results from this round of sampling showed normal resin acid concentrations. We conclude that high concentrations are probably rare.

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